PRODUCTION OF ZINC IRON PHOSPHATE GLASSES BY MICROWAVE AND CONVENTIONAL PROCESSING METHODS

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ABSTRACT

This thesis describes the production of zinc iron phosphate glasses, xZnO-(40-x)Fe_2O_3-60P_2O_5 with x = 0, 2.5, 5.0, 7.5, 10.0 (mol%) using microwave and conventional processing methods. The work aiming to investigate the potential use of microwave processing method for the production of zinc iron phosphate glasses. In microwave glass melting, magnetite powder coupled with microwave radiation within 10 minutes radiation time. Equivalent samples are conventionally prepared by melting the identical batches at 1300 ˚C for 3 hours in electric furnace. The mass loss trends for all samples are increased with the increasing of ZnO and in agreement with the theoretical data calculated from batch compositions. It is found that the mass loss of microwave samples increased about 2 % compared to conventionally made samples and this is due to the contribution of Fe^{2+} ion. XRD patterns confirmed the amorphous structure and revealed an identical diffuse scattering behaviour of all samples. The homogeneity of chemical compositions are evident from SEM images and the chemical analysis using EDS implied that the percentage amount of Zn, Fe and P in each of the glasses prepared using microwave method decrease compared to the samples formed using conventional method. The T_g data are in range of 459 – 476 ˚C and these agree with the mass loss and EDS analyses. Although the chemical properties of conventionally prepared samples are superior compared to the microwaved samples, the powder density analysis shown that the microwave samples possessed higher powder density values (3.0491, 3.0488 and 3.0343 g/cm^3) at lower ZnO content (< 5 %). All in all, it is clear from the data that the microwave glass melting are promising and viable method as it retains high density of materials, rapid glass melting and reduce energy consumption compared to conventional glass melting.

ABSTRAK

Tesis ini menerangkan penghasilan gelas zink ferum fosfat, xZnO-(40-x)Fe_2O_3-60P_2O_5, x = 0, 2.5, 5.0, 7.5, 10.0 (mol%) menggunakan kaedah pemprosesan gelombang mikro dan konvensional. Kajian ini bertujuan mengkaji potensi kaedah pemprosesan gelombang mikro dalam penghasilan gelas zink ferum fosfat. Semasa proses penghasilan gelas menggunakan gelombang mikro, magnetit telah berjaya berganding dengan sinaran gelombang mikro dalam masa 10 minit. Sampel yang sama dihasilkan secara konvensional melalui kaedah peleburan gelas dalam relau elektrik pada suhu 1300 ˚C selama 3 jam. Trend kehilangan jisim bagi semua sampel menunjukkan peningkatan selari dengan ZnO dan keadaan ini sepadan dengan data teori yang dikira berdasarkan komposisi kumpulan bahan. Kehilangan jisim bagi sampel gelombang mikro telah meningkat sebanyak 2 % berbanding sampel yang dihasilkan secara konvensional dan ini disebabkan oleh peningkatan ion Fe^{2+}. Corak XRD mengesahkan kewujudan struktur amorfus dan keadaan taburan selerak yang sama bagi semua sampel. Keseragaman komposisi kimia terbukti melalui imej SEM dan analisis kimia menggunakan EDS menunjukkan jumlah peratusan Zn, Fe dan P dalam gelas yang dihasilkan menggunakan kaedah pemprosesan gelombang mikro menurun berbanding sampel yang dihasilkan menggunakan kaedah konvensional. Data T_g menunjukkan keputusan di dalam julat 459 – 476 ˚C dan keadaan ini selari dengan keputusan analisis kehilangan jisim dan EDS. Walaupun ciri kimia bagi sampel yang dihasilkan secara konvensional lebih baik berbanding sampel yang dihasilkan melalui kaedah pemprosesan gelombang mikro, analisis ketumpatan serbuk menunjukkan sampel gelombang mikro mempunyai kadar ketumpatan serbuk yang lebih tinggi (3.0491, 3.0488 and 3.0343 g/cm^3) pada kandungan ZnO yang lebih rendah (< 5 mol%). Kesimpulannya, proses peleburan gelas menggunakan gelombang mikro adalah kaedah yang berdaya maju kerana mampu mengekalkan kadar ketumpatan bahan yang tinggi, proses yang pantas serta mampu mengurangkan penggunaan tenaga berbanding peleburan gelas secara konvensional.

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LIST OF SYMBOLS AND ABBREVIATIONS

ε	-	Dielectric
m	-	Mass
P	-	Pressure
T_m	-	Melting temperature
T_g	-	Glass transition temperature
V	-	Volume
DMO	-	Domestic microwave oven
DTA	-	Differential thermal analysis
EDS	-	Energy dispersive X-ray
Fe^{2+}	-	Iron (II) ion
Fe^{3+}	-	Iron (III) ion
FeO	-	Iron (II) oxide
Fe_2O_3	-	Iron (III) oxide
Fe_3O_4	-	Iron (II,III) oxide, Magnetite
FTIR	-	Fourier transform infrared spectroscopy
$NH_4H_2PO_4$	-	Ammonium dihydrogen phosphate
P_2O_5	-	Phosphorus pentoxide
RT	-	Room temperature
SEM	-	Scanning electron microscopy
XRD	-	X-ray diffraction
Zn^{2+}	-	Zinc ion
ZnO	-	Zinc oxide

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CHAPTER 1

INTRODUCTION

1.1 Research background

Phosphate glasses can be formed by melting and quenching single element of phosphorus pentoxide, P_2O_5 or mixed with other chemical components depending on the targeted applications. The advantages of phosphate glasses such as low processing temperature < 1150 ˚C, high thermal expansion coefficient and low softening temperature (Jermoumi *et al.*, 2002) made it suitable for engineering application *i.e.* fast ion conducting materials (Das, Srivastava & Singh, 2012), laser host materials (Sene, Martinelli & Gomes, 2004), glass to metal seals (Shrikhande, 2009) and immobilisation of nuclear wastes (Day *et al.*, 1998). The main issue of most phosphate glasses is that it can reacts with water at ambient temperature and leads to overall poor chemical durability. This issue can be overcome by introducing iron oxides in the phosphate glass composition (Reis, Karabulut & Day, 2001; Jermoumi *et al.*, 2002; Li *et al.*, 2013; Li, Lu & Yang, 2014). It is clear in the literature that Fe_2O_3-P_2O_5 glasses could achieved high chemical durability better than alkali-doped iron phosphate glasses and free from toxic materials (Li, Yang & Ren, 2013). The addition of ZnO into phosphate glass (ZnO-Fe_2O_3-P_2O_5) is of interest in this work as ZnO has the ability to decrease the glass melting temperature, which is beneficial to sealing application (Li *et al*., 2013).

The most common method used to produce zinc iron phosphate glasses is conventional processing involving heat transfer mechanisms of conduction, convection and/or radiation using standard electric furnace. In this case, the surface of the raw materials will be heated from outside to inside of the sample; heat propagated from the heating element of the furnace to the surface of the sample. The existence of the thermal loss occurred by transferring the heat at multiple

mediums causing the production of phosphate glasses to be less economical and typically requires at least 5 hours melting time at around 1100 ˚C. This drawback encourages researchers to find new glass processing method that is more efficient in heating and overall cost effective. Therefore, in this work, an alternative processing method using microwave radiation is studied and aiming to form identical properties of glasses to that conventional processing method.

The microwave processing method for the production of zinc iron phosphate glasses offers several advantages compare to the conventional processing method. For example, the processing time of glasses can be reduced from hours to minutes thus the energy consumption and the cost of preparing the zinc iron phosphate glasses can be significantly reduced (Mayzan *et al.*, 2014, 2015). To make useful direct microwave radiation in the production of glass, it is essential for the composition to include at least one microwave absorber component that can couple to the microwave field at frequency range of 900 MHz and 2.45 GHz; once coupled with microwave radiation, the heat will be generated from the molecular levels and heat up very rapidly (Rao *et al.*, 1999; Kharissova *et al.*, 2010). In this research, Fe_3O_4 (microwave absorber) is selected as heating source for synthesising zinc iron phosphate glasses by microwave radiation. The physical, chemical, thermal and structural properties of zinc iron phosphate glasses prepared using microwave and conventional processing methods are compared systematically.

1.2 Problem statements

The energy consumption for producing zinc iron phosphate glasses using conventional processing method is not efficient due to high thermal losses via conduction, convection and/or radiation heat transfer mechanisms. The presence of thermal gradient also contributed to slow the overall glass melting process of which requires at least 5 hours melting time at temperature around 1100 ˚C. In conventional heating, slow heating rates are selected to reduce the steep thermal gradient thus leading to process-induced stress (Das *et al.*, 2008). Currently, microwave processing method has been highly promoted by some researches as improved heating technology of which decreasing the processing time and

increasing the rate of chemical reaction (Almeida, Martinelli & Partiti, 2007; Chenu, Lebullenger & Rocherullé, 2010; Hémono *et al.*, 2010; Mayzan *et al.*, 2014; Renuka *et al.*, 2016). This attracts the interest to utilise microwave processing method in the production of zinc iron phosphate glasses. To the best of knowledge, production of zinc iron phosphate glasses by microwave processing method is still not being reported in literature.

Previous studies found that the glasses prepared using the microwave processing method tend to have a better/similar properties to the glasses prepared using the conventional processing method (Mandal, Balaji & Sen, 2014; Mandal *et al.*, 2013; Prasad *et al.*, 2010). This characteristic is due to the higher heating and diffusion rates which allowed improvements in physical and mechanical properties of the glasses produced owing to the lower formation of defects. Renuka *et al.* (2016) stated that when iron oxides (Fe_3O_4) coupled with microwave radiation, it can be reduced and/or oxidised depending on the heating environment and glass composition thus altering the physical and structural properties of the glasses. However, to this date, the study on the properties of the zinc iron phosphate glasses prepared using the microwave processing method is still not widely discovered and worth further investigation.

1.3 Significance of research

The study on microwave processing method for the production of zinc iron phosphate glasses will benefit current glass industries as it is fast, clean and capable to produce identical properties of glasses that are prepared using conventional processing method. Microwave heating occurs at molecular levels and heat propagates via rapid volumetric heating. This reduces the processing time and energy consumption as well as producing homogeneous glass materials. An identical melting temperature can be achieved through microwave heating by manipulating the amount of microwave absorber such as iron oxides. The property of iron oxides are interesting as it can interacts with both electric and magnetic field of microwaves. When iron oxides couple with the microwave radiation, the heat will be generated from the molecular levels and thus heating up the sample

very rapidly. This study will clarify the use of iron oxides as microwave absorber element.

1.4 Aim and objectives of study

The aim of this research is to investigate the potential use of microwave processing method for the production of zinc iron phosphate glasses. The aim can be divided into three objectives as follow:

i. To synthesise zinc iron phosphate glasses using microwave processing method;
ii. To synthesise zinc iron phosphate glasses using conventional processing method;
iii. To characterise and compare the physical, chemical, thermal and structural properties of zinc iron phosphate glass prepared using microwave and conventional glass processing methods.

1.5 Scope of study

The scope of the research is limited to the mentioned objectives of which considering glass compositions of xZnO-(40-x)Fe_2O_3-60P_2O_5 where x = 0, 2.5, 5.0, 7.5, 10.0 (mol%). The samples are only prepared using the microwave and conventional processing methods. In microwave processing, a domestic microwave oven (Panasonic, model NN-ST342M) is consistently used throughout the experiment whilst for the conventional processing, a box electrical furnace (Protherm, model PLF 130/15) is used to produce equivalent sample to that microwave processing. The samples were investigated by using various analysis/characterisation methods including assessing mass loss, powder X-ray diffraction (XRD), scanning electron microscope (SEM) equipped with energy-dispersive X-ray spectroscopy (EDS), differential thermal analysis (DTA), powder densities and Fourier transform infrared spectroscopy (FTIR).

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

In this chapter, a literature survey on glass materials, phosphate glasses, application of zinc iron phosphate glass, processing routes for the production of glass materials, production of zinc iron phosphate glasses using conventional processing method, previous studies of glass melting using microwave processing method as well as the impact of utilising microwave processing method in glass industry are presented and discussed.

2.2 Glass materials

Glasses can be defined as materials that are produced by any techniques, have an amorphous structure, absent of long-range order of the crystalline structure and exhibit glass transition behaviour (Shelby, 2005; Mayzan, 2015; Tyagi & Banerjee, 2017). Essentially, glass materials prepared using oxide chemical compounds are known as oxide glasses. The constituents of oxide glasses can be divided into three categories; network formers (*i.e.* P_2O_5, SiO_2, B_2O_3), intermediates (*i.e.* Al_2O_3, PbO, ZnO) and modifiers (*i.e.* Li_2O, Na_2O, CaO). Network former is the main constituent in the glass materials of which has the ability to form glass on its own. In the case of network intermediates, these chemical compounds might function as network formers or modifiers depending on the glass composition. Sometimes, intermediate oxide is being added in high volume for linking up with the basic glass network to retain structural continuity. The network modifiers present as ions to break up or alter the glass network and thus reducing the glass network connectivity (Tyagi & Banerjee, 2017); the bridging oxygen compensated by non-bridging

oxygen in oxide glasses. Bridging oxygen are oxygen atoms that links units together whilst non-bridging oxygen are oxygen atoms that are not shared between units. Oxide glasses can be composed from the combination of at least one network former with or without a modifier and/or intermediate components. The structural property of oxide glass obeyed the rules of Zachariasen's model; the oxygen atom must linked not more than two glass former atoms, the coordination number of the glass former is small, the oxygen polyhedral share corners with each other and linked in three-dimensional network (Zachariasen, 1932).

An oxide glass can be prepared by melting suitable mixture of raw material(s) as above and undergone cooling process of which causes the viscosity of the undercooled melt to grow several orders of magnitude over a small temperature interval. The time spent on cooling is not enough for the atoms to re-arrange in order to attain an equilibrium structure (crystalline structure). The glass transition happened when the system is said to have driven out of equilibrium. This phenomena can be further understood from Figure 2.1 which illustrated the comparison of volume versus temperature for glass and crystalline solids.

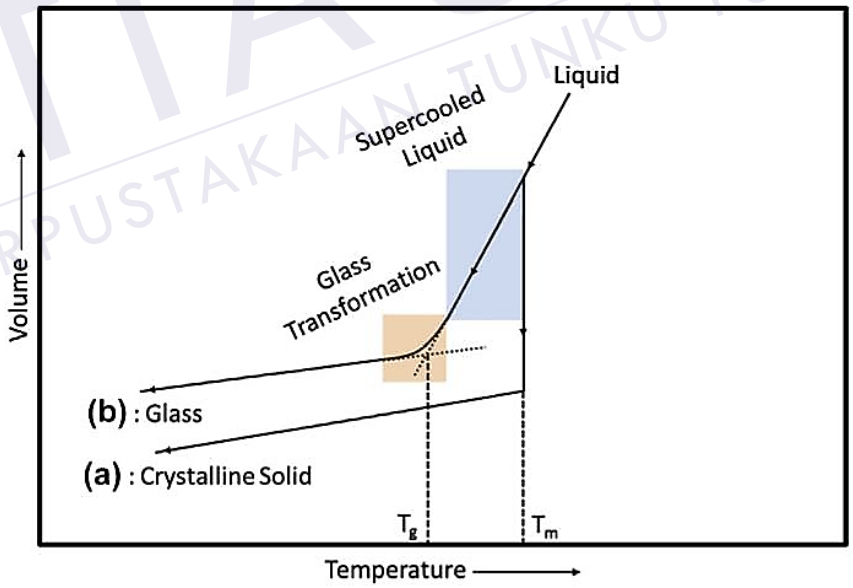

Figure 2.1: Variation of volume versus temperature to form (a) crystalline solid (b) glass (taken from Tyagi & Banerjee, 2017)

Based on the figure, there are two possible conditions exist when the temperature reached the melting point, T_m,; crystallisation and supercooling. For the case of crystallisation, it took place when there is a discontinuous change in

volume at T_m whilst for supercooling, the liquid state is maintained below T_m and this is known as a supercooled liquid (blue region in Figure 2.1). The transformation from liquid state to supercooled liquid can be regarded as the first step in the formation of glass. As the supercooled liquid undergoes further cooling, the viscosity increases rapidly and reached maximum viscosity. At this stage, the liquid started to solidify and eventually form a glass (Tyagi & Banerjee, 2017). The glass transition region is the point where the liquid state changes from liquid-like to solid-like glass (orange region in Figure 2.1). Specifically, the glass transition temperature, T_g, is the temperature at the intersection point of the tangents between the liquid state and glass curves.

2.3 Phosphate glasses

As mentioned previously in section 1.1, the phosphate glasses are widely used as fast ion conducting materials (Das *et al.*, 2012) and laser host materials (Sene *et al.*, 2004). This is due to their properties *i.e.* good electrical properties, high thermal expansion coefficient, low melting and softening temperatures (Jermoumi *et al.*, 2002). However, the practical applications of the phosphate glasses are often limited due to their poor chemical durability. This limitation can be improved by the addition of one or more oxides such as Fe_2O_3 (Reis *et al.*, 2001), ZnO (Matori *et al.*, 2015), and SnO (Bhat, Ganguli & Rao, 2003). The addition of these oxides enables the replacement of the P-O-P bond with a more moisture resistant bond and thus improving the overall chemical durability of the glass (Yu *et al.*, 1997). Previous study by Matori *et al.* (2015) prevailed that addition of Zn^{2+} ions caused changes in the cross-link density thus overall enhances the density of the glass produced. Theoretically, the cross-link density can be understood as the density of chains that connect two infinite parts of the glass network.

In general, the basic structural units of phosphate glasses are PO_4 tetrahedra. The tetrahedra are normally classified using the Q^i notation, where *i* represents the number of bridging oxygen per tetrahedron. The networks of phosphate glasses can be categorised depending on the O/P ratio from the glass composition. The possible variations of tetrahedral in phosphate type glasses are presented in Figure 2.2. A Q^0 is related to the isolated tetrahedral (orthophosphate group, O/P=4), Q^1 to end

groups (pyrophosphate, O/P=3.5), Q^2 to middle groups (metaphosphate, O/P=3) and Q^3 to branching group (ultraphosphate, O/P=2.5) (Brow, 2000; Li *et al.*, 2013). The structure of the vitreous P_2O_5 consists of Q^3 tetrahedral with a three-dimensional cross-linked network. Hence, by adding the network modifier to the P_2O_5, the phosphate structural groups may deduced from ultraphosphate Q^3 to metaphosphate Q^2 to pyrophosphate Q^1 to orthophosphate Q^0 respectively (Pascuta *et al.*, 2010).

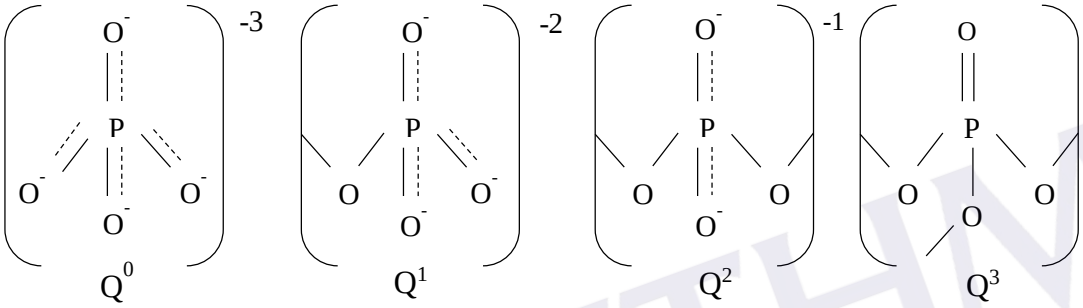

Figure 2.2: Phosphate tetrahedral sites that may exist in phosphate glasses (Brow, 2000)

2.3.1 Iron phosphate glasses

The element of iron in phosphate glass has been well-known to significantly increase the chemical durability. The addition of iron oxide, Fe_2O_3 to the phosphate glass composition replaced the P-O-P bond with more hydration resistant P-O-Fe^{2+} or/and P-O-Fe^{3+} bonds (Reis *et al.*, 2001). As a result, iron phosphate glasses are of great interest for the immobilisation of high-level nuclear wastes containing high amount of sulphates (Bingham & Hand, 2008) and chrome oxides (Huang *et al.*, 2004). Among the various iron phosphate glass compositions, the glass with $60P_2O_5$-$40Fe_2O_3$ (mol%) is evident to be more chemically durable (Day, Ray & Kim, 2004). Apart from their excellent chemical durability, much lower viscosity iron phosphate glass has a melting temperature of 100-200 K lower and homogenisation time at 1.5-3 hours shorter compared to the borosilicate glass (Kim & Day, 2003). Also, these unique properties made iron phosphate glass a good candidate to incorporate/encapsulate any hazardous wastes that possessing huge amount of volatile elements.

2.3.2 Zinc iron phosphate glasses

The improvement of chemical durability decreased the thermal stability and increased the T_g of glasses. However, the mentioned condition is less favourable for the glasses to be applied in the applications of glass sealant. This is due to the fact that sealing frits with poor thermal stability tend to crystallise during cooling or reheating treatment processes thus hindered the viscous flow and affecting the sealing performance (Li *et al.*, 2017).

The addition of zinc oxide, ZnO in iron phosphate glass composition is promising as it contains no toxic materials and is found to be more durable than alkali doped iron phosphate glass (Li *et al*., 2013). In agreement with other study, the presence of the zinc oxide reduced the T_m and T_g of the zinc iron phosphate glass (Jain *et al.*, 2009; Li *et al.*, 2013; Li *et al.*, 2017). The general structure (see Figure 2.3) of the zinc iron phosphate glass can be visualised as PO_4 tetrahedra joined together by oxygen polyhedral containing Fe^{2+} or Fe^{3+} and Zn^{2+} ions in interstitial positions of which act as a modifier in the glass network (Reis *et al.*, 2001). The typical properties of zinc iron phosphate glasses with a range of composition from 0-40 (mol%) of ZnO, 0-40 (mol%) of Fe_2O_3 and 50-60 (mol%) P_2O_5 are listed in Table 2.1.

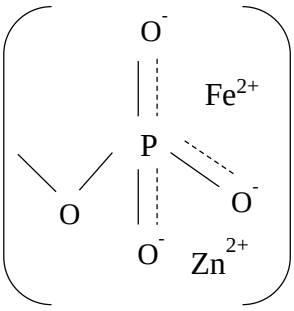

Figure 2.3: Example of general structure zinc iron phosphate glass (Zinc;Iron(2+);Phosphate, 2007)

Table 2.1: Typical properties of zinc iron phosphate glass

Property	Description	Reference
Physical		
Colour	- Shiny black	(Saha, 2010)
Density	- ranging from 2.76 to 3.50 g/cm^3 - increased with increasing Fe_2O_3 content	(Reis *et al.*, 2001; Jermoumi *et al.*, 2002; Li *et al.*, 2013; Li *et al.*, 2013)
Melting temperature	- 1000-1250 ˚C	(Li *et al.*, 2013)
Chemical		
Crystalline phase	- Amorphous phase	(Reis *et al.*, 2001; Jermoumi *et al.*, 2002; Saha, 2010; Li *et al.*, 2013, 2017)
Thermal		
Glass transition temperature, T_g	- ranging from 404 ˚C to 473 ˚C	(Li *et al.*, 2013; Li *et al.*, 2017, 2013; Saha, 2010)
Structural		
Chemical bond formation	- See Figure 2.4 for the example of FTIR spectra of the zinc iron phosphate glass - contain large number of Fe(II)-O-P and Fe(III)-O-P bond in the region of wavenumber from 500 cm^{-1} to 1320 cm^{-1}	(Reis *et al.*, 2001; Jermoumi *et al.*, 2002)

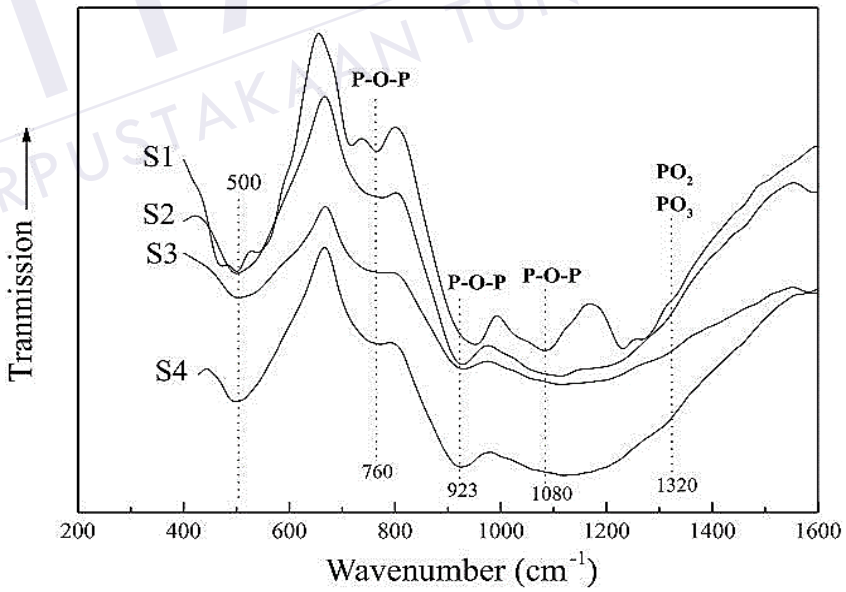

Figure 2.4: FTIR spectra for zinc iron phosphate glasses (taken from Reis *et al.*, 2001)

2.3.2.1 Mass loss

Previous studies have calculated the theoretical mass loss data for the $40Fe_2O_3$-$60P_2O_5$ (mol%) glasses prepared using microwave and conventional processing methods based on the chemical equation considering two situations; all of the iron turns to Fe^{2+} or Fe^{3+} ion (Mayzan *et al*., 2014). Mayzan *et al.* (2014) have found that all of the iron in the microwaved glasses present as Fe^{2+} ion and the experimental mass loss data for the glasses prepared using the microwave processing method was higher than the ones produced conventionally.

Almeida *et al.* (2007) prevailed that the glasses produced in electric furnace have higher Fe^{3+} ion content whilst the ones produced in microwave oven have higher Fe^{2+} ion content. The iron in the glasses produced in the microwave oven underwent reduction. The reason being related to the short time for melting or by a change of the local atmosphere (Almeida *et al.*, 2007).

2.4 Sealing glass frits application

Glass frits are finely powdered glasses that when re-heated will sinter, soften and flow to form a seal. Historically, the lead based glass frits are the most common glass sealant however lead jeopardise safety and health issues as it is toxic and hazardous to the biological environment (Nian *et al.*, 2018; Saha, 2010). Zinc iron phosphate glasses have been suggested as an alternative for the sealing application due to their low T_g, comparable chemical durability to the iron phosphate glass and its thermal stability is near similar to the widely used soda lime silica glass (Morena, 2000; Zhang *et al.*, 2008).

In sealing application, zinc iron phosphate glass frits are frequently used to seal soda lime silica glass windshield (Saha, 2010), incandescent lamps, float glass (Brow *et al.*, 1997) and gaseous discharge devices (Wei, Hu & Hwa, 2001). There are two types of classification for glass frits; vitreous and devitrifying types. Vitreous type frits are non-crystallising glasses which will remain glassy throughout the sealing cycle and can be reworked repeatedly without leaving any marks or layers on the subject, while the devitrifying type frits crystallize during the curing cycle and form a two phase glass-ceramic material, which is usually

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